AFRL-AFOSR-VA-TR-2016-0040

# Shape Descriptors for the Quantification of Microstructures

MARC DeGRAEF
CARNEGIE MELLON UNIVERSITY

01/05/2016
Final Report



Air Force Research Laboratory
AF Office Of Scientific Research (AFOSR)/ RTB1
Arlington, Virginia 22203
Air Force Materiel Command

# REPORT DOCUMENTATION PAGE

*Form Approved*
*OMB No. 0704-0188*



| | | |
|---|---|---|
| **1. REPORT DATE** *(DD-MM-YYYY)*<br>14-01-2016 | **2. REPORT TYPE**<br>Final Performance | **3. DATES COVERED** *(From - To)*<br>30-09-2012 to 29-09-2015 |

| | |
|---|---|
| **4. TITLE AND SUBTITLE**<br>Shape Descriptors for the Quantification of Microstructures | **5a. CONTRACT NUMBER** |
| | **5b. GRANT NUMBER**<br>FA9550-12-1-0475 |
| | **5c. PROGRAM ELEMENT NUMBER**<br>61102F |
| **6. AUTHOR(S)**<br>MARC DeGRAEF | **5d. PROJECT NUMBER** |
| | **5e. TASK NUMBER** |
| | **5f. WORK UNIT NUMBER** |
| **7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)**<br>CARNEGIE MELLON UNIVERSITY<br>5000 FORBES AVENUE<br>PITTSBURGH, PA 15213-3815 US | **8. PERFORMING ORGANIZATION REPORT NUMBER** |
| **9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)**<br>AF Office of Scientific Research<br>875 N. Randolph St. Room 3112<br>Arlington, VA 22203 | **10. SPONSOR/MONITOR'S ACRONYM(S)**<br>AFRL/AFOSR RTB1 |
| | **11. SPONSOR/MONITOR'S REPORT NUMBER(S)** |

**12. DISTRIBUTION/AVAILABILITY STATEMENT**
A DISTRIBUTION UNLIMITED: PB Public Release

**13. SUPPLEMENTARY NOTES**

**14. ABSTRACT**
The main objective of this research project was to establish an accurate methodology for the quantification of 3-D shapes in both experimental and
synthetic microstructures in two-phase materials, as well as an accurate comparison metric between them. Furthermore, this project should establish
a mathematical and numerical methodology for the analysis and identification of 3-D shapes based on random 2-D sections and explore ways in
which this shape information can be correlated to materials properties. The research was carried out by the PI and two graduate students, Lily
Nguyen and Ryan Harrison. The grant provided support for the students (tuition+stipend) and the PI (Summer salary) as we as travel support to
attend conferences and review meetings. The program has benefitted from interactions with external groups at OSU, UCSB, UCLA, UMich and
the Karlsruhe Institute of Technology (KIT, Germany). The report provides research highlights organized by published/pending papers.

**15. SUBJECT TERMS**
image analysis, microstructure

| **16. SECURITY CLASSIFICATION OF:** | | | **17. LIMITATION OF ABSTRACT** | **18. NUMBER OF** |
|---|---|---|---|---|
| | | | | |

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

| a. REPORT | b. ABSTRACT | c. THIS PAGE | | PAGES | 19a. NAME OF RESPONSIBLE PERSON |
|---|---|---|---|---|---|
| Unclassified | Unclassified | Unclassified | UU | | MARC DeGRAEF |
| | | | | | 19b. TELEPHONE NUMBER *(Include area code)* |
| | | | | | 412-268-8527 |

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

DISTRIBUTION A: Distribution approved for public release.

# Shape Descriptors for the Quantification of Microstructures

Final Report submitted by
Carnegie Mellon University
to the
Air Force Office of Science and Research
Grant # FA-95501210475

AFOSR/Dr. A. Sayir
Directorate of Aerospace, Chemistry
and Materials Science
875 North Randolph Street
Suite 325, Room 3026
Arlington, VA 22203

*Principal Investigator*
M. De Graef

December 20, 2015

# 1. Program Overview

The main objective of this research project was to establish an accurate methodology for the quantification of 3-D shapes in both experimental and synthetic microstructures in two-phase materials, as well as an accurate comparison metric between them. Furthermore, this project should establish a mathematical and numerical methodology for the analysis and identification of 3-D shapes based on random 2-D sections and explore ways in which this shape information can be correlated to materials properties.

The research was carried out by the PI and one graduate student, Lily Nguyen, who graduated with a Ph.D. on Friday May 1, 2015; Lily is currently working at the Naval Research Laboratory as a post-doctoral researcher in the group of Dr. David Rowenhorst. The AFOSR grant provided financial support for Lily (tuition+stipend) and the PI (Summer salary support) as well as travel support to attend conferences and review meetings. A new graduate student, Ryan Harrison, joined the group in the late Fall of 2014, and worked in parallel with Lily until she graduated; Ryan has continued the shape descriptor work and we will summarize some of his most recent work in a later section. Since the AFOSR program ended in September 2015, Ryan has started working on the AFOSR-MURI program.

The program benefitted from research interactions with several groups outside CMU. We interacted with Prof. Yunzhi Wang (OSU) and his graduate students and published two joint papers. Lily visited Prof. Tresa Pollock's group (UCSB) to learn about superalloys, and interacted with Dr. Alan Ardell (UCLA) to compare their shape analysis of super alloy $\gamma'$ particles with our own. She also collaborated with Prof. Veera Sundararaghavan (UMich) and his group on the quantitative evaluation of shapes in synthetically generated microstructures. Finally, Lily obtained a 2013-2014 International Research Fellowship from the ICMR program at UCSB; she worked at the Karlsruhe Institut für Technologie (KIT) in Karlsruhe, Germany, for several weeks in collaboration with Prof. Peter Gumbsch and his group.

In the following sections we highlight research results from this program by citing chronologically each of the papers that were published with program support as well as one paper that was published during the current program with support of the previous AFOSR grant.

# 2. Selected Research Highlights

## 2.1. **Morphological aspects of materials structures.** This section is based on the following paper:

> P.G. Callahan, J.P. Simmons, and M. De Graef, "A quantitative description of the morphological aspects of materials structures suitable for quantitative comparisons of 3D microstructures," Modelling Simul. Mater. Sci. Eng. **21** (2013) 015003 (doi:10.1088/0965-0393/21/1/015003).[1]

While many laboratories can produce 3D models of microstructure from serial sectioning or tomography, the more widespread practice is to attempt to infer 3D shapes from 2D sections of different orientations. We have developed a forward modeling approach that, given a 3D particle shape, produces statistically representative 2D sections whose projections are characterized by 2D moment invariants (MI). Since the sectioning plane is random, each particle will produce a statistical distribution of 2D moments and an ensemble of particles in a microstructure will produce its own characteristic distribution of 2D moments and can be used as a representation of the microstructure. This distribution can be represented as a point in a metric space. We use the Hellinger distance as the measure for this space, which allows us to quantify the similarity of two microstructures. Example applications include: determination of a 3D shape by computing the Hellinger

[1]This paper was initiated with support under a previous AFOSR grant (FA-95500710179) but publication was only completed under the current grant.

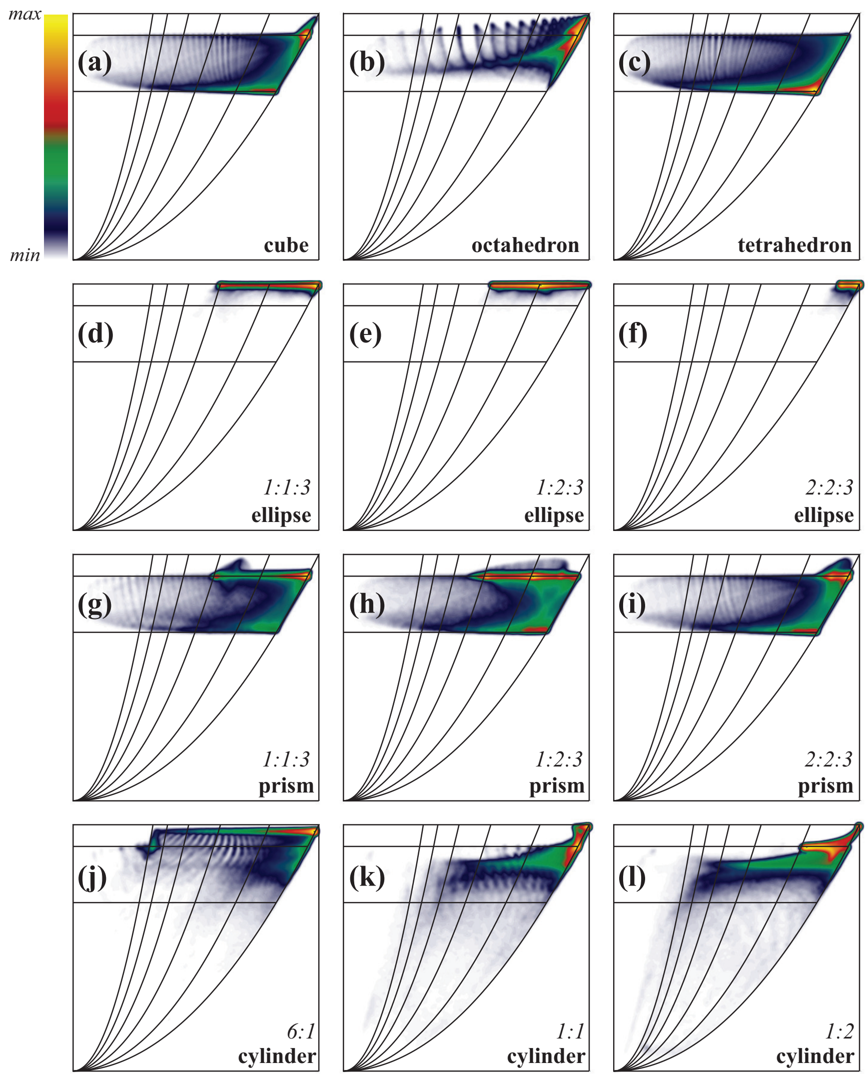



FIGURE 1. Second-order moment invariant density maps (SOMIM) for the following shapes: (a) cube, (b) octahedron, (c) tetrahedron, (d)–(f ) ellipses with aspect ratios 1 : 1 : 3, 1 : 2 : 3 and 2 : 2 : 3, (g)–(i) rectangular prisms with the same aspect ratios, and (j )–(l) circular cylinders with height-to-diameter ratios 6 : 1, 1 : 1 and 1 : 2. The striations in the low amplitude regions are due to the voxelized nature of the object. Solid horizontal lines indicate the geometric locations of the prisms and triangles; parabolas indicate shapes of constant aspect ratio. The color legend is shown at the top left.

distance between MI density maps derived from random 2D section micrographs and the density map database; automated detection and quantification of rafting in cuboidal microstructures; and quantitative comparison of pairs of microstructures.

One of the novel applications of this paper is the introduction of the Second Order Moment Invariant Map (SOMIM) and the Projected Moment Invariant Map (PMIM) which, taken together, provide a comprehensive way to classify 2D shapes or 2D intersections of 3D shapes. Fig. 1 shows the SOMIM plots for a series of 3D shapes, indicating that each 3D shape has its own SOMIM (and PMIM) fingerprint. Comparisons between shape maps can then be made quantitatively by using the Hellinger distance, $H(p, q)$ between two normalized distributions (histograms) $p$ and $q$,

defined as:

$$H(p,q) = \left(1 - \sum_{i=1}^{N} \sqrt{p_i q_i}\right)^{\frac{1}{2}}$$

where each distribution has $N$ bins $p_i$ or $q_i$. This distance provides a true metric for comparison of distributions; the smaller the value of $H$, the more similar the two distributions are.

2.2. **The abnormal strain state in ferroelastic materials.** This section is based on the following paper:

L. Nguyen, D. Wang, Y. Wang, and M. De Graef, "Quantifying the abnormal strain state in ferroelastic materials: a moment invariant approach," Acta Materialia, **94**, 172-180 (2015) (doi: 10.1016/j.actamat.2015.04.057).

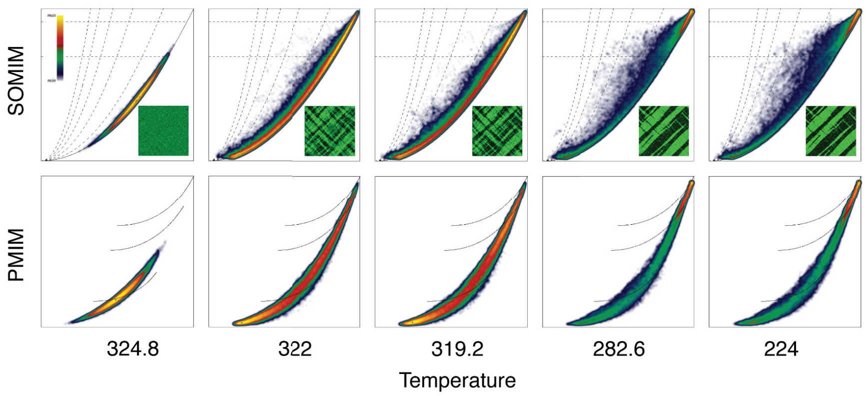



FIGURE 2. The SOMIM and PMIM density maps of a system undergoing a martensitic transformation in the presence of a point defect concentration of $c = 0.025$, with decreasing temperature (in K). This series of plots shows a transformation from the parent phase (T=324.8 K) to the pre-martensitic tweed (T=322, 319.2K) to martensite (T=224 K).

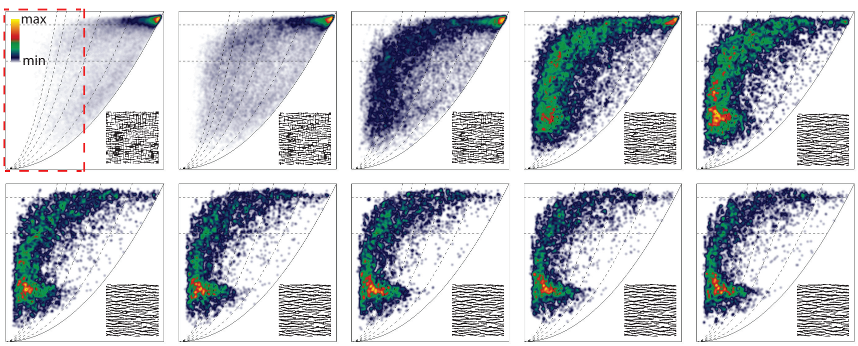
The strain glass transition has been found in many ferroelastic systems, but the microstructural nature of strain glass is still unclear. Here, two-dimensional second and fourth order moment invariants as well as image entropy are used to evaluate the presence of a strain-glass state in simulated microstructure images of a doped ferroelastic system. Four different microstructural states are identified, depending on the doping concentration of point defects and the temperature. The martensitic state is characterized by a broad moment invariant distribution peaking near the invariants for the circle, whereas systems that display the strain glass transition produce distinctly different distributions. The image entropy is found to increase with increasing defect concentration; above the critical defect concentration of 0.1, image entropy becomes nearly linearly dependent on temperature. The image analysis approach is capable of characterizing the range of strain domain shapes that occur in the different microstructural states of a doped ferroelastic system.

The results in Fig. 2 show the SOMIM (top row) and PMIM (bottom row) maps computed for the inset microstructures, where the black and green colors indicate the microstructure variants. It is clear that the appearance of a tweed microstructure has a profound effect on the shape descriptor maps, and that diffuse intensity appears to the side of the main parabola (which corresponds to isotropic shapes). These maps were computed by moving a circular window across the microstructure and plotting the corresponding moment invariants for each window location on the SOMIM and PMIM histograms. This figure clearly illustrates that moment invariant shape descriptors are capable of detecting and capturing the subtle microstructural changes that occur when small-scale modulations modify the microstructure. We have also shown that image entropy can provide an alternative sensitive detector for such changes.

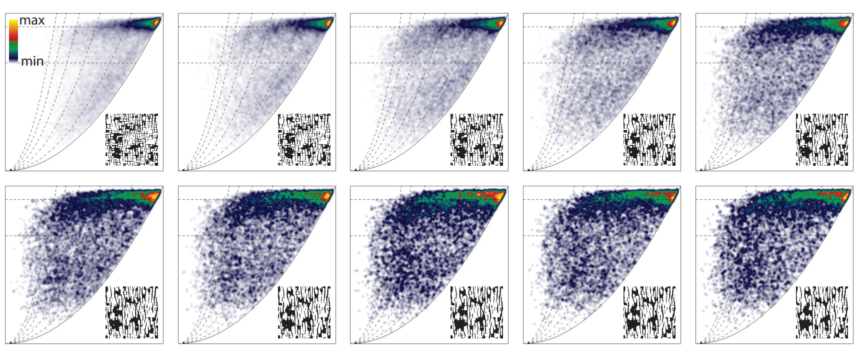
**2.3. Rafting of $\gamma'$ precipitates in superalloys.** This section is based on the following paper:

L. Nguyen, R. Shi, Y. Wang and M. De Graef, "Quantification of rafting of $\gamma'$ precipitates in Ni-based superalloys," Acta Materialia, **103**, 322-333 (2016) (doi: 10.1016/j.actamat.2015.09.060)

Rafting, the directional coarsening of $\gamma'$ precipitates in a $\gamma/\gamma'$ microstructure, can affect the mechanical performance and lifetime of Ni-based superalloys. While rafting has been commonly observed in many systems, a criterion for the onset and completion of rafting has not been fully established. In this work, we employ 2D and 3D moment invariants (MIs) to track the evolution of a synthetic $\gamma/\gamma'$ microstructure under a tensile load as a function of time. Systems with negative and positive misfit are considered. Under normal conditions, $\gamma'$ precipitates have high MI values characteristic of cuboidal shapes, while rafted precipitates have low MI values. The onset of rafting is defined as the time at which more than half of the $\gamma'$ precipitates have 2D MIs in particular regions of interest of the MI density maps. The rafting completion time is defined as the time at which the initial linear trend slows down with time. The MI approach is also compared to other shape parameters and is shown to provide a convenient all-in-one shape descriptor that can detect the elongation and wavy morphology of the rafted precipitates, as well as the morphological changes in the $\gamma/\gamma'$ microstructure as a function of time.




(a) SOMIM - negative misfit




(b) SOMIM - positive misfit

FIGURE 3. SOMIM density maps for the negative (a) and positive (b) misfit microstructures for ten time intervals; 256 images per time interval were analyzed. The region of interest used to determine rafting completion is shown in the red boxes in the first map of (a) and (b). The peak of the MI distributions shifts from the upper right corner to the lower left corner as the $\gamma'$ precipitates directionally coarsen.

In this paper, we have shown clearly (see Fig. 3) that moment invariants, and in particular the SOMIM density map, can be used to identify the onset of rafting, even when there are no obvious

microstructural changes visible to the human eye. The moment invariants provide sensitive shape descriptors, and we have reported on both 2D and 3D analysis approaches.

2.4. **On the use of odd-order moment invariants.** Third and higher order odd moments have proven to be more difficult to use in shape recognition and image analysis. The main hurdle to be overcome is the vanishing of these odd order moments for shapes which are symmetric about the origin, such as those commonly seen in material microstructures. Palaniappan et al.[2] suggested the use of a shift factor in both the $x$ and $y$ directions to be used in the central moments calculation :

$$x_s = \sqrt{\frac{\mu_{20}}{\mu_{00}}}; \quad y_s = \sqrt{\frac{\mu_{02}}{\mu_{00}}},$$

leading to the following shifted moments:

$$\mu^s_{pq} = \int_{-\infty}^{\infty}\int_{-\infty}^{\infty}(x - \bar{x} + x_s)^p(y - \bar{y} + y_s)^q.$$

If one considers a shape already centered at the origin (that is $\bar{x} = \bar{y} = 0$), then these shifted moments can simply be written in terms of the moments of the original unshifted shape ($\mu_{pq}$):

$$\mu^s_{pq} = \sum_{k=0}^{p}\sum_{l=0}^{q}(-1)^{q-l}\binom{p}{k}\binom{q}{l}(x_s)^{p-k}(y_s)^{q-l}\mu_{kl}.$$

Because the odd order moments for a centrosymmetric shape are equivalent to zero, this sum becomes a combination of the even order moments of the shape multiplied by the shift factor, itself based upon even order moments.

Thus, the shifted odd order moments of a shape, while nonzero for symmetric objects, do not actually provide any additional information about the object that was not already included in the original central moments. This does not mean they are completely useless, however. When analyzing distributions of shapes which contain both symmetric and non-symmetric shapes, the range of traditional central moment invariants of the third order is very large. The shift allows for the values of these invariants for the circle to be used as normalization factors, which limit the range of these values considerably and allows their use in the calculation of invariants. The use of odd-order moment invariants will continue to be explored as part of the ongoing MURI research program.

2.5. **Principal Component Analysis and moment invariants.** By utilizing the above coordinate shift when performing moment invariant calculations, moment values can be determined for any order and for any shape. Utilizing previous second and fourth order moment invariants, and also including six third order moments, we have a total of eleven moment invariants to consider. Therefore, each 2D shape can be thought of as being represented by a point in an eleven dimensional space defined by these values. In order to visualize such a space, a non-arbitrary projection to a lower-dimensional space is required. Utilizing principal component analysis (PCA) allows one to calculate the axes which account for the largest amount of variation in the data and to assign a proportion of the variation to each. By mapping the data to the two or three principal components which explain the majority of the variation, one can reduce the dimensionality to a more easily visualized state while retaining as much information as possible.

Utilizing PCA in the creation of shape maps allows for a wider range of shape information to be displayed in a single image. Figure 4 shows the difference between a SOMIM and a PCA map for two experimental superalloys. This illustrates the power of the PCA approach in combination with a variety of shape parameters. The SOMIMs do not allow for easy differentiation between the two alloys because both of the peaks are in nearly the same location and the spread is small.

[2] R. Palaniappan, P. Raveendran and S. Omatu, Improved Moment Invariants for Invariant Image Representation, Invariants for Pattern recognition and Classification (World Scientific Publishing Co., Singapore, 2000), pp. 167–187.

However, the PCA maps spread the data across a larger region and the differences between the two distributions are much more noticeable. However, due to the nature of the axes of the PCA maps, some interpretability is lost. This will be remedied in the future by identifying regions associated with specific classes of shapes. This is an area of ongoing research.

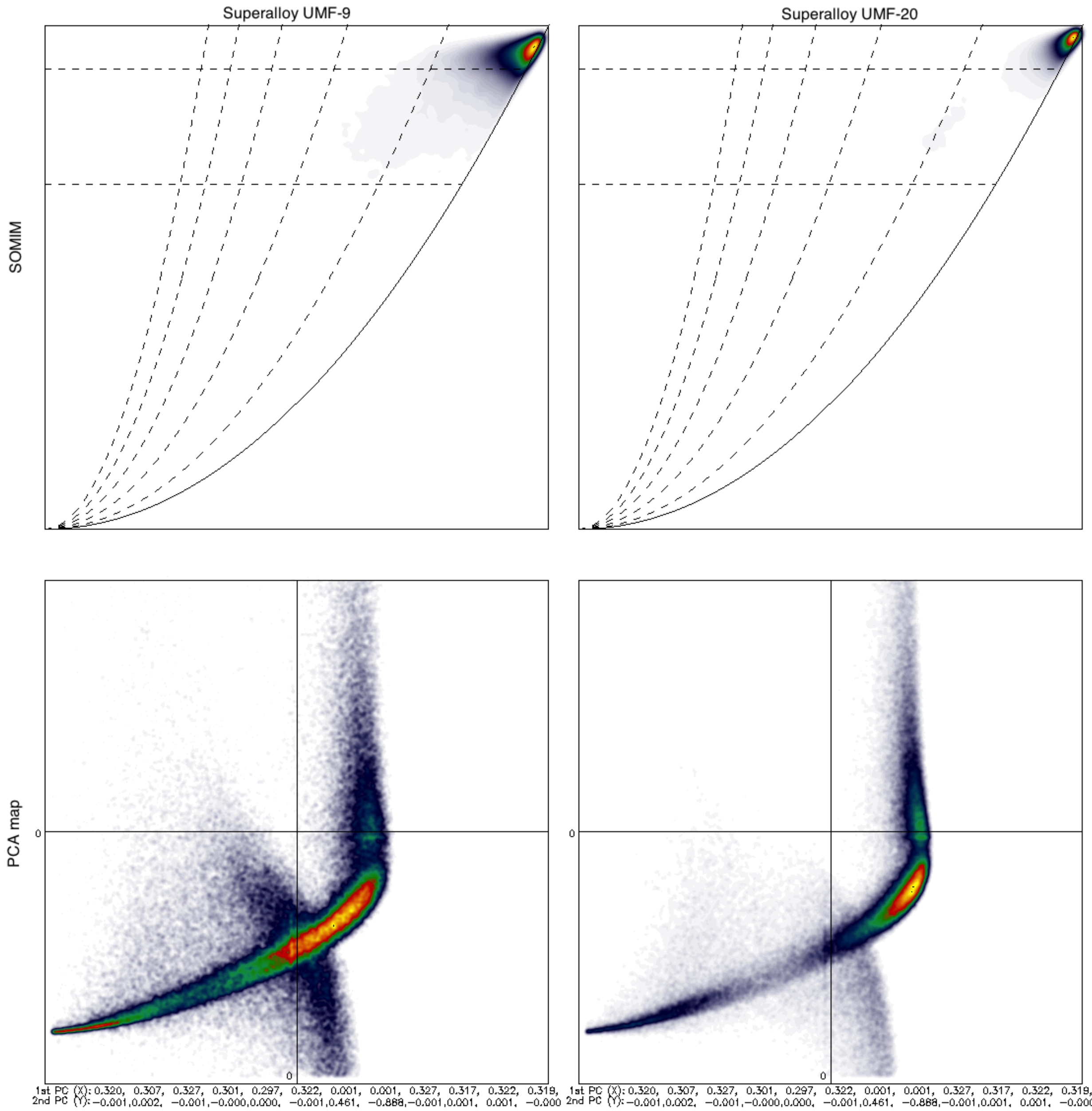



FIGURE 4. Comparison of SOMIM and PCA maps between two experimental superalloy microstructures. The two principal components used retain 75.2% of the total variation in the data. The coefficients of the invariants for each PC are shown. They are in order: two second order, six third order, and three fourth order invariants.

Archival publications (published or pending) during reporting period:

(1) P.G. Callahan, J.P. Simmons, and M. De Graef, "A quantitative description of the morphological aspects of materials structures suitable for quantitative comparisons of 3D microstructures," Modelling Simul. Mater. Sci. Eng. **21** (2013) 015003 (doi:10.1088/0965-0393/21/1/015003).

(2) L. Nguyen, D. Wang, Y. Wang, and M. De Graef, "Quantifying the abnormal strain state in ferroelastic materials: a moment invariant approach," Acta Materialia, **94**, 172-180 (2015) (doi: 10.1016/j.actamat.2015.04.057).

(3) L. Nguyen, R. Shi, Y. Wang and M. De Graef, "Quantification of rafting of $\gamma'$ precipitates in Ni-based superalloys," Acta Materialia, **103**, 322-333 (2016) (doi: 10.1016/j.actamat.2015.09.060)

(4) "Nondestructive evaluation of 3D microstructure evolution in strontium titanate," M. Syha, W. Rheinheimer, L. Nguyen, B. Loedermann, W. Augustin, A. Trenkle, W. Ludwig, D. Weygand, M. De Graef, M.J. Hoffmann, and P. Gümbsch, Acta Mater. (under review, 2015)

(5) "Towards a Quantitative Comparison between Experimental and Synthetic Grain Structures," P.G. Callahan, M. Groeber, and M. De Graef, Acta Materialia, (submitted, 2015).

Changes in research objectives, if any: None

Change in AFOSR program manager, if any: None

Extensions granted or milestones slipped, if any: None

Include any new discoveries, inventions, or patent disclosures during this reporting period (if none, report none): None

1.

**1. Report Type**

Final Report

**Primary Contact E-mail**
**Contact email if there is a problem with the report.**

degraef@cmu.edu

**Primary Contact Phone Number**
**Contact phone number if there is a problem with the report**

412-268-8527

**Organization / Institution name**

Carnegie Mellon University

**Grant/Contract Title**
**The full title of the funded effort.**

Shape Descriptors for the Quantification of Microstructures

**Grant/Contract Number**
**AFOSR assigned control number. It must begin with "FA9550" or "F49620" or "FA2386".**

FA9550-12-1-0475

**Principal Investigator Name**
**The full name of the principal investigator on the grant or contract.**

Marc De Graef

**Program Manager**
**The AFOSR Program Manager currently assigned to the award**

Dr. Ali Sayir

**Reporting Period Start Date**

10/01/2012

**Reporting Period End Date**

09/30/2015

**Abstract**

The main objective of this research project was to establish an accurate methodology for the quantification of 3-D
shapes in both experimental and synthetic microstructures in two-phase materials, as well as an accurate comparison metric between them. Furthermore, this project should establish a mathematical and numerical methodology for the analysis and identification of 3-D shapes based on random 2-D sections and explore ways in which this shape information can be correlated to materials properties.

The research was carried out by the PI and one graduate student, Lily Nguyen, who graduated with a Ph.D. on Friday May 1, 2015; Lily is currently working at the Naval Research Laboratory as a post-doctoral researcher in the group of Dr. David Rowenhorst. The AFOSR grant provided financial support for Lily (tuition+stipend)
and the PI (Summer salary support) as well as travel support to attend conferences and review meetings. A new graduate student, Ryan Harrison,
joined the group in the late Fall of 2014, and worked in parallel with Lily until she graduated; Ryan has continued the shape descriptor work and we will summarize some of his most recent work in a later section. Since the AFOSR program ended in September 2015, Ryan has started working on the AFOSR-MURI

program.

The program benefitted from research interactions with several groups outside CMU. We interacted with Prof. Yunzhi Wang (OSU) and his graduate students and published two joint papers. Lily visited Prof. Tresa Pollock's
group (UCSB) to learn about superalloys, and interacted with Dr. Alan Ardell (UCLA) to compare their shape analysis
of super alloy gamma' particles with our own. She also collaborated with Prof. Veera Sundararaghavan (UMich)
and his group on the quantitative evaluation of shapes in synthetically generated microstructures. Finally, Lily
obtained a 2013-2014 International Research Fellowship from the ICMR program at UCSB; she worked at the Karlsruhe
Institut fur Technologie (KIT) in Karlsruhe, Germany, for several weeks in collaboration with Prof. Peter Gumbsch and
his group.

**Distribution Statement**

**This is block 12 on the SF298 form.**

Distribution A - Approved for Public Release

**Explanation for Distribution Statement**

**If this is not approved for public release, please provide a short explanation. E.g., contains proprietary information.**

**SF298 Form**

**Please attach your SF298 form. A blank SF298 can be found here. Please do not password protect or secure the PDF The maximum file size for an SF298 is 50MB.**

AFD-070820-035.pdf

**Upload the Report Document. File must be a PDF. Please do not password protect or secure the PDF . The maximum file size for the Report Document is 50MB.**

FinalReportDec2015.pdf

**Upload a Report Document, if any. The maximum file size for the Report Document is 50MB.**

**Archival Publications (published) during reporting period:**

(1) P.G. Callahan, J.P. Simmons, and M. De Graef, "A quantitative description of the morphological aspects of materials structures suitable for quantitative comparisons of 3D microstructures," Modelling Simul. Mater. Sci. Eng. 21 (2013) 015003 (doi:10.1088/0965-0393/21/1/015003).
(2) L. Nguyen, D. Wang, Y. Wang, and M. De Graef, "Quantifying the abnormal strain state in ferroelastic materials: a moment invariant approach," Acta Materialia, 94, 172-180 (2015) (doi: 10.1016/j.actamat.2015.04.057).
(3) L. Nguyen, R. Shi, Y. Wang and M. De Graef, "Quantification of rafting of γ′ precipitates in Ni-based superalloys," Acta Materialia, 103, 322-333 (2016) (doi: 10.1016/j.actamat.2015. 09.060)
(4) "Nondestructive evaluation of 3D microstructure evolution in strontium titanate," M. Syha, W. Rheinheimer, L. Nguyen, B. Loedermann, W. Augustin, A. Trenkle, W. Ludwig, D. Weygand, M. De Graef, M.J. Hoffmann, and P. Gu¨mbsch, Acta Mater. (under review, 2015)
(5) "Towards a Quantitative Comparison between Experimental and Synthetic Grain Structures," P.G. Callahan, M. Groeber, and M. De Graef, Acta Materialia, (submitted, 2015).

**Changes in research objectives (if any):**

None

**Change in AFOSR Program Manager, if any:**

None

**Extensions granted or milestones slipped, if any:**

None

**AFOSR LRIR Number**

**LRIR Title**

**Reporting Period**

**Laboratory Task Manager**

**Program Officer**

**Research Objectives**

**Technical Summary**

**Funding Summary by Cost Category (by FY, $K)**

| | Starting FY | FY+1 | FY+2 |
|---|---|---|---|
| Salary | | | |
| Equipment/Facilities | | | |
| Supplies | | | |
| Total | | | |

**Report Document**

**Report Document - Text Analysis**

**Report Document - Text Analysis**

**Appendix Documents**

## 2. Thank You

**E-mail user**

Dec 20, 2015 06:49:00 Success: Email Sent to: degraef@cmu.edu